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Improvement of laser deposited high alloyed powder metallurgical tool steel by a post-tempering treatment

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Abstract

Laser cladding process of a high alloyed powder metallurgical tool steel was studied for die repairing purposes. The low hardness obtained after the deposition process was improved by later tempering cycles, achieving crack free coatings with hardness well above 700 HV. The effect of different post tempering cycles was investigated in order to determine the optimal temperature range. The microstructure of the samples was studied using optical and scanning electron microscope and the volumetric ratio of retained austenite was determined by X-ray diffraction. The tempering effect was mainly evaluated through cross-section microhardness profiles.

Keywords: powder metallurgy; tool steels; laser cladding; Nd:YAG laser

1. Introduction

Over the last years, the introduction of new materials like the High Strength Steels in the automotive forming industry has led the tool manufacturers to design new tools to overcome the more and more aggressive working conditions in the processing of these materials. In this frame, new tools made of powder metallurgical tool steels are starting to be used by the toolmakers. These special steels offer an excellent combination of toughness, hardness and wear resistance. In particular, the HWS Isotropic, which is a cold work tool steel, has been identified as an excellent material for bending dies.

Nevertheless, there is a lack of knowledge about the repair behavior of these materials, and a concise study is needed to reach the full exploitation of their potential. Specifically, the need to identify new

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repair processes is crucial from an economic point of view for the complete exploitation of these materials in the tooling field.

The laser cladding technique arises as a good alternative since it provides a good metallurgical bonding with the substrate offering deposits of coating material with wear, corrosion and high-temperature oxidation resistance that guarantees the toughness of the base material [1]. Concerning the application of this process to high alloyed powder metallurgical tool steels, some research studies can be found in the state of the art [2-7]. The mechanical properties achieved when depositing different materials by laser cladding are highly dependent on the chemical composition of the coating material. Very hard coatings may be produced when depositing some of these powder metallurgical tool steels like CPM 10V [2-3] or Vanadis 4 Extra [5], which may even be improved by subsequent heat treatments in some cases [6]. Nevertheless, other materials present a rather low hardness after the laser deposition process (as-clad), and subsequent heat treatments are required in order to improve their mechanical properties.

The aim of this work is therefore to demonstrate the suitability of the combination of laser cladding and subsequent tempering cycles to produce HWS Isotropic layers with similar properties to the hardened substrate.

2. Experimental procedure

Heat treated HWS Isotropic plates (63-65 HRc) with a thickness of 20 mm were used as the substrate and the same material in gas atomized state was used as the coating metal, which presents a spherical shape and a particle size of 45-90 μm, as it can be observed in Fig. 1. The chemical composition of this material, obtained by chemical analysis, is shown in Table 1. The substrate surfaces were machined before laser treatment as in a conventional repair operation.

Table 1. Chemical composition (wt. %) of the studied material

Material	C	Cr	Mn	Mo	Si	V	W
HWS Isotropic	1.08	7.80	0.34	1.86	1.38	2.66	1.73

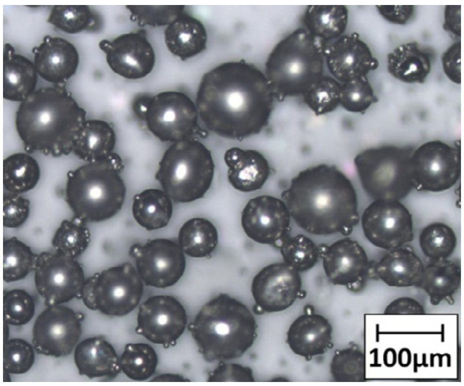

Fig. 1. Gas atomized HWS Isotropic powder particles

Table 2. Processing parameters used in the deposition of the coatings

Laser power (KW)	1.2
Scanning speed (mm/s)	15
Powder flow rate (g/min)	5
Gas pressure (bar)	2
Overlap (%)	35

The laser cladding process was conducted using a 2.2 kW diode pumped continuous wave Nd:YAG laser. The laser beam was guided to the working area by a 0.6 mm diameter circular fibre and a laser head with an optical system, able to provide a defocused 2.7 mm circular spot with Gaussian-like energy profile in the working-area. A powder injection system was used to deliver the coating powder material into the molten pool through a discrete laser cladding nozzle. Helium was used as the coaxial shielding gas to prevent surface oxidation, fixing the pressure at 2 bar. The substrate was preheated to 250 ºC in order to prevent cracking of the coating or the substrate [7]. Single layer multi-track coatings with 35 % overlap ratio were carried out obtaining 0.5 mm thick layers, covering an area of 60 x 15 mm. After a comprehensive parameter search, the processing parameters shown in Table 2, were chosen as the optimum for the coatings analyzed in the present work.

Post-tempering cycles at different temperatures were carried out in an oven. These cycles consisted on three different stages, each of 2 hours, starting at 20 ºC below the set temperature in the first stage, following at 10ºC below the set temperature in the second stage and reaching the defined set temperature in the third stage. The workpiece was air-cooled down to room temperature between each stage.

Microstructural analyses were carried out on transverse sections by optical and scanning electron microscopy (SEM). For this analysis the samples were polished with diamond powder and chemically etched in a solution of HNO_3:HCl:H_2O with a volumetric proportion of 1:2:3. Semi-quantitative chemical analysis was performed in SEM by means of energy dispersive spectroscopy (EDS).

Microhardness measurements were performed in the same transverse sections, using a Vickers hardness tester with a load of 0.1 kg in the centre of the central laser cladding track, from the surface to the material core (vertical) and across the clad zone, parallel to the original surface at 0.2 mm above it (horizontal).

An X-ray diffractometer with parallel beam (2 mm diameter pinhole) and Cr radiation, operated at 40 kV and 40 mA was used to determine the volume fraction of retained austenite, following ASTM E975-3 standard [8]. Retained austenite measurements were done at the centre of the surface of each coating. Due to the low penetration of X-rays, the information obtained comes from the very surface, around 5-10 µm deep.

3. Results and Discussion

3.1. Microstructure

The cross sections of the coatings present three regions of interest (Fig. 2): the clad zone (CZ), which is the region where the melting has taken place, including the coating and the dilution zones, the re-quenched zone (RQZ), which is the region of the base metal in which temperature above the austenization point was achieved without reaching the melting point, and the heat affected zone (HAZ), in which enough temperature to affect the original microstructure was reached, but without austenization.

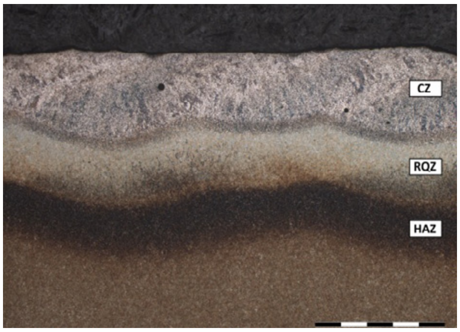

Fig. 2. Optical macrograph showing the different zones of the HWS Isotropic coatings (CZ: Clad Zone, RQZ: Re-Quenched Zone, HAZ: Heat Affected Zone)

The CZ of the as-deposited sample presents a cellular structure (Fig. 3), with rod-like carbides gathered within the grain boundaries, and a martensitic matrix with a considerable amount of retained austenite. The EDS patterns measured in these rod-like carbides reveal the existence of all the carbide forming elements present in the alloy composition, with a major content of chromium and vanadium. This indicates that mixed type carbides have precipitated in the grain boundaries. A similar carbide distribution was observed when depositing a layer of Vanadis 4 Extra by this technique [5]. In the regions in which no melting has taken place (RQZ and HAZ), the carbides remain unaltered, exactly as they can be observed in the base material. Two types of carbides are detected: The largest ones, with a size of roughly 1-2 μm, are rich in chromium, while the smallest ones, of less than 0.5 μm in size, are mainly vanadium carbides, according to the EDS patterns obtained in both types of carbides. Therefore, the main difference of the RQZ and the HAZ with respect to the base material lies not in the carbides but in the metal matrix. In the case of the RQZ, temperatures above the austenization critical one have been achieved which, along with the rapid cooling rate associated with the process, leads to a quenching process, resulting in a matrix of quenched martensite plus some retained austenite. With regard to the HAZ, the temperature was not high enough to re-austenize the matrix, but the martensite of the base material was tempered.

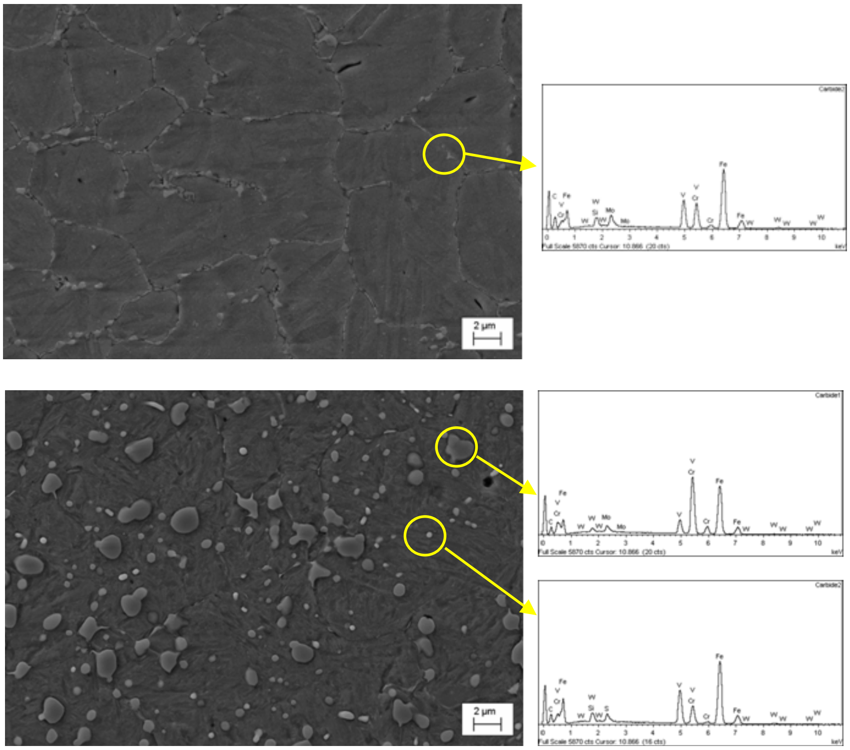

Fig. 3. SEM micrograph of the as deposited HWS Isotropic coating Clad Zone (top) and the base material (bottom) with the EDS patterns measured in different carbides

The two possible effects that may lead to the secondary hardening of the HWS Isotropic coatings after post tempering cycles were studied: the precipitation of secondary carbides and the transformation of the retained austenite into tempered martensite. The SEM micrographs show no differences whatsoever in carbide number, size or shape, unless a tempering temperature of 650 ºC or above is employed. When the tempering process is carried out at those high temperatures, lots of tiny carbides (less than 0.2 µm) start to precipitate (Fig 4) depleting the matrix in carbon. Although carbides are harder, the matrix of tempered martensite with less amount of carbon presents a lower hardness, thus decreasing the overall hardness of both the coating and the base material, as will be shown later in paragraph 3.2, Fig 6.

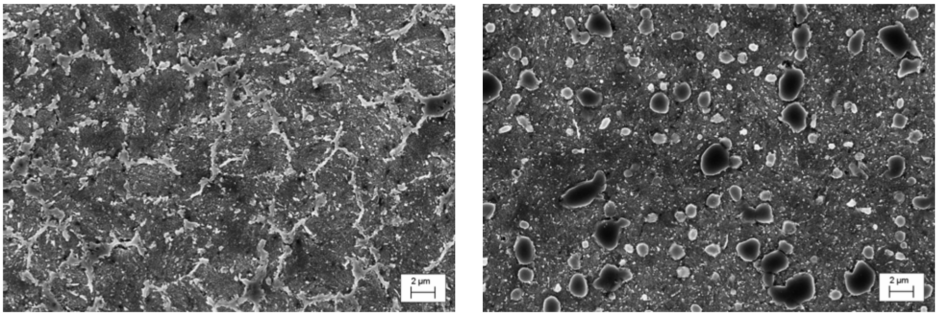

Fig. 4. SEM Micrograph of the HWS Isotropic coating Clad Zone (left) and the base material (right) after a post-tempering cycle at 750 ºC

Table 3. Calculated volumetric fractions of retained austenite after post-tempering cycles at different temperatures

	As deposited	350 ºC	550 ºC	650 ºC	750 ºC
Retained Austenite Fraction (vol %)	11.4	4.1	1.5	0.0	0.0

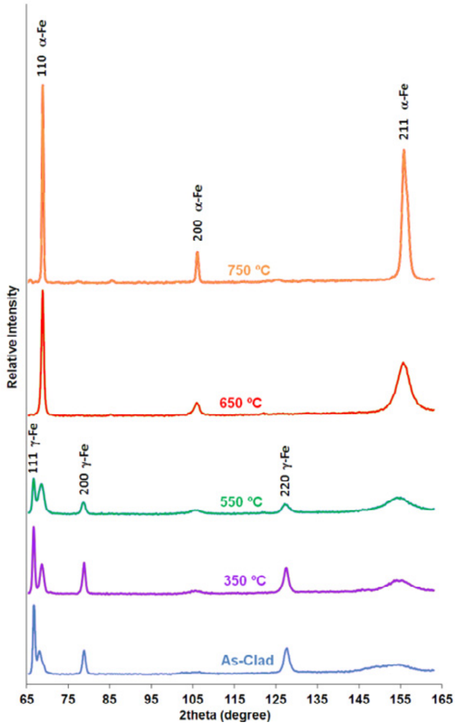

Fig. 5. X-ray diffraction patterns of the samples after post-tempering cycles at different temperatures

Both the X-ray diffraction patterns shown in Fig. 5 and the calculated values of Table 3 reveal a gradual reduction of the volumetric fraction of the retained austenite when increasing the tempering temperature. Although a considerable amount of retained austenite can still be found in the sample tempered at 350 ºC, there is almost none in the coating tempered at 550 ºC and it is completely transformed when tempering at temperatures above 650 ºC.

Taking into account these two effects, best hardness results are expected at a temperature of 550 ºC, due to the low austenite content achieved without depleting the metal matrix of carbon by the precipitation of new carbides.

3.2. Microhardness

Microhardness profiles measured in the vertical (from the surface of the coating to the base material core) and horizontal (parallel to the original surface, at 0.2 mm above it) directions are shown in Fig. 6. A hardness of roughly 600 HV0.1 is achieved in the CZ of the as-deposited coating, 200HV0.1 lower than the original base material hardness. The horizontal profile reveals a high heterogeneity across the coating, which is caused by the multiple thermal cycles that take place in each region of the coating due to the necessity of depositing various tracks in order to cover wide areas. With regard to the effect of the deposition process in the base material, slightly lower hardness than the original one is observed in the RQZ of the as-clad coating, probably due to some retained austenite that results from the re-quenching process. In the case of the HAZ, the martensite of the original microstructure is tempered, lowering its hardness, especially in the upper regions, in which the highest tempering temperatures were achieved.

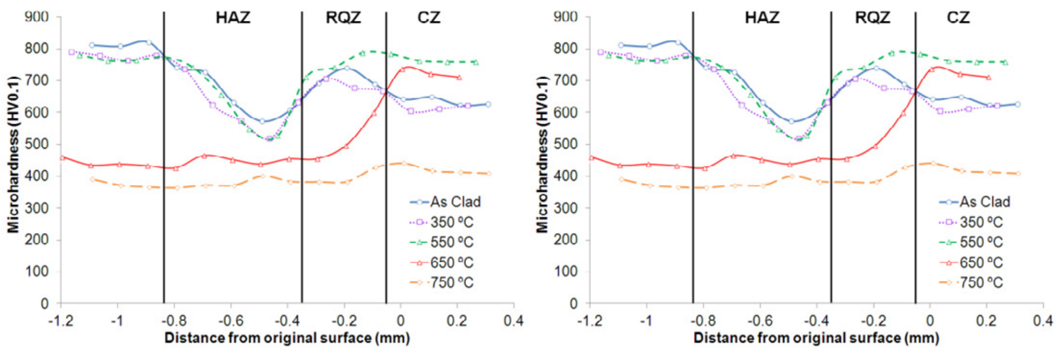

Fig. 6. Vertical (left) and horizontal (right) microhardness profiles measured after tempering cycles at different temperatures

No significant change of hardness is observed in any region when tempering at 350 ºC, as it can be observed in both hardness profiles (Fig. 6), which means that this temperature was not high enough to produce any noteworthy change of microstructure.

The best results are obtained when tempering at 550ºC, as expected in accordance with the microstructure analysis. Not only a similar hardness as the base material is achieved in the CZ, but it is also much more uniform than the original as-deposited profile. The hardness of the RQZ is also slightly improved when tempering at 550 ºC, which confirms the hypothesis of certain amount of austenite being retained in that region in the as-deposited condition. The hardness of the HAZ could not be improved due to the fact that the high temperature tempered martensite of that region can only be affected by even higher temperatures, and in that case the effect would be detrimental, as it can be observed in the hardness profiles of the samples tempered at 650 and 750 ºC.

A certain improvement of hardness is still observed in the CZ of the coating tempered at 650ºC, probably due to the transformation of the retained austenite. Nevertheless, the hardness of deeper zones is highly lowered, in particular the hardness of the base metal drops to a value below 500 HV0.1 which is unacceptable in the case of a real repairing process of a hard tool.

Finally, a tempering temperature of 750 ºC is proven to be too high, obtaining a uniform hardness of about 400 HV0.1 both in the coating and the base material.

4. Summary and conclusions

Coatings of HWS Isotropic were produced by laser cladding and tempered in an oven at different temperatures in order to investigate the effect of the post-tempering temperature on the microstructure and microhardness of different zones, and therefore to study the viability of using a post-tempering treatment to improve the hardeness of the coating. The study was centered not just on the coating but in every affected zone of the base material as well.

The microstructure of the as-deposited coating, or clad zone, is formed of rod-like carbides gathered in the grain boundaries of a martensite plus retained austenite matrix. A re-quenched zone is observed just below the clad zone in which the carbides of the base material were not affected, but some retained austenite is detected in the re-quenched martensitic matrix. Finally a region is observed in which the martensite of the base material is tempered by the deposition process.

The as-deposited heterogeneous hardness value of roughly 600 HV0.1, can be improved and homogenized by choosing the correct tempering temperature. A temperature of 350 ºC is not high enough to affect the structure of any of the studied regions and thus no variation of hardness is observed in the sample tempered at this temperature. A tempering temperature of 550 ºC allows improving the hardness of the coating up to approximately 750 HV0.1, which is a value almost as high as that of the original base metal. Furthermore, a more uniform profile is observed in this sample in comparison with the as-deposited condition. Nonetheless, special care has to be taken, in order to not exceed this tempering temperature, since higher values are proven to be harmful, highly lowering the hardness of the coating and even of the base material.

These results demonstrate that the hardness of the laser deposited coatings of HWS Isotropic can be highly improved by subsequent tempering cycles at the correct temperature, without negatively affecting the base material, hence validating the combination of laser cladding plus a subsequent tempering process to repair tools made of this material. Further studies will include the possibility to reproduce the post-tempering process by using the same laser employed for the cladding process instead of an oven.

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References

[1] Toyserkani, E.; Khajepour, A.; Corbin S.: Laser cladding. CRC Press, 2005.

[2] Hu, Y. P.; Chen, C. W.; Mukherjee K.: Development of a new laser cladding process for manufacturing cutting and stamping dies. In: Journal of materials science 33 (1998), 1287-1292.

[3] Wang, S. H.; Chen, J. Y.; Xue, L.: A study of the abrasive wear behaviour of laser-clad tool steel coatings. In: Surface & Coatings Technology 200 (2006), 3446-3458.

[4] Stasko, W.; Pinnow, K. E.; Dixon, R. B.: Proceeding of the 5th International Conference on Advanced Particulates materials and Process, Metal Powder Industries federation, Princeton, NJ,1997, 401.

[5] Leunda J.; Soriano C.; Sanz C.; García Navas V.: Laser cladding of vanadium-carbide tool steels for die repair. In: Physics Procedia 12 (2011), 345-352.

[6] Chen, J. Y.; Wang, S. H.; Xue, L.: On the development of microstructures and residual stresses during laser cladding and post-heat treatments. In: Journal of Materials Science 47 (2012), 779-791.

[7] Zhang Y.; Yuan X.; Zeng X.: ICALEO'01, proceedings, Sec. D, 705.

[8] ASTM E 975-03. Standard Practice for X-Ray Determination of Retained Austenite in Steel with Near Random Crystallographic orientation. ASTM Internacional, 2003.